

was detected in these blank determinations that we were not certain whether it came from the Soluene or other sources. You may note that the blood of control rats which was analyzed with the use of Soluene did not contain detectable amounts of asbestos and zero fibers per gram was reported.

We have since repeated this work (as yet unpublished) in an experiment in which asbestos was fed to rats and the tissues analyzed by a low temperature ashing technique similar to that reported for fecal asbestos (2). Essentially similar results were obtained as in the original work with significantly higher levels of asbestos fibers found in treated than in control animals.

H. M. CUNNINGHAM  
R. D. PONTEFRACT

Health Protection Branch  
Health and Welfare Canada  
Ottawa, Ontario KIA 0L2, Canada

1. Pontefract, R. D., and Cunningham, H. M. *Nature*, 243: 352 (1973).
2. Cunningham, H. M., Pontefract, R. D., and O'Brien, R. C. J. *Toxicol. Environ. Health* 1: 377 (1976).

Dear Sir:

Polychlorinated biphenyls (PCBs), ubiquitous environmental pollutants, are usually assayed by gas chromatography of cleaned-up extracts with electron capture detection. Quantitation is relative to some arbitrary standard, usually one of the commercial PCB mixtures (Aroclors, Clophens, etc.). The mixtures recovered from environmental samples (soil, feces, various animal tissues, milk and the like) almost never match the PCB standard mixture in composition, but the commercial mixture best approximating the unknown in general distribution of peaks on the chromatograms is necessarily selected as quantitation reference.

Although it is well known (1,2) that electron capture detectors respond differently to different PCBs, the assumption is usually made that the er-

rors in comparing an unknown mixture with a more-or-less similar standard mixture will cancel out. Accordingly, the literature is replete with data on "PCB content" of all sorts of environmental samples (3). In a few cases (4), the numbers reported were roughly confirmed by perchlorination to the single compound decachlorobiphenyl (which can be accurately quantitated with an electron capture detector), but in the vast majority of cases, there has been no confirmation.

To illustrate the unreliability of the more common direct gas chromatography of PCBs versus an Aroclor standard, we performed the following simple experiment. A 1-g portion of Aroclor 1260 was chromatographed on 50 g of Florisil PR, eluting with light petroleum ether. Under these conditions, the PCBs tend to "tail". The portion eluting between 200 and 300 ml of petroleum ether weighed about 4 mg and showed all of the same peaks as the original Aroclor 1260 during gas chromatography. However, the relative proportions of the peaks differed from those of stock 1260.

Three preparations were supplied for gas chromatographic analysis; the original Aroclor 1260, for use as reference standard, cottonseed oil spiked with 11.0  $\mu$ g Aroclor 1260/g oil, and cottonseed oil spiked with 10.0  $\mu$ g Aroclor 1260 "tail" (simulated environmental sample)/g oil. The analyst was simply instructed to analyze the two cottonseed oil samples for "total PCB content", using the Aroclor 1260 as reference standard. He did not know what PCB mixture or what concentration range to expect to find in the oil (i.e., a single-blind experiment).

Work-up of the samples was typical of procedures used for fatty materials, involving extraction into hexane:benzene, 5:1 (v/v), partitioning with sulfuric acid to remove lipids, drying with sodium sulfate-sodium carbonate mixture, and chromatography on Florisil. In every case, elution (with hexane:diethyl ether, 94:6) was shown to be sufficient to remove all of the PCBs from the column (no PCBs were seen in a subsequent elution with hexane:diethyl ether, 85:15). Gas chromatography was routine, with a Varian 2100 gas chromatograph,  $\text{Sc}^3\text{H}$  electron capture detector, and a glass column (2 mm ID  $\times$  6 ft) of 1.5% OV-17 + 1.95% QF-1 on 80/100 Gas Chrom Q. Each sample was analyzed five times. Quantitation was done in two ways: by summing all of the peak areas attributable to PCBs, and by summing the areas of four conspicuous peaks selected from chromatograms of the Aroclor 1260 standard. Both methods are commonly used in different

laboratories.

The analyst reported that "Sample B" (the oil spiked with Aroclor 1260) contained  $10.4 \pm 1.7 \mu\text{g}$  PCB/g oil. Thus, his procedure gave a recovery of 94.5% of the PCB spike (the cottonseed oil had previously been found to be free of PCBs at the 10 ppb level). For the 1260 "tail" sample, he found a PCB content of  $3.53 \pm 1.03 \mu\text{g/g}$  oil using total peak areas, and  $3.06 \pm 0.66$  using four major peaks. Thus, the analytical results were low by about a factor of three.

We used the high molecular weight 1260 to avoid the risk of evaporative losses, and, since the variation of electron capture response among isomers is least for the PCBs having more than four chlorine atoms (1), to bias the analytical results in favor of as little error as possible. Thus, we believe that this experiment casts doubts upon the reliability of all literature data on "PCB content" of environmental samples, whenever the

sample analyzed did not exactly match the reference standard in distribution of components and an electron capture detector was used for quantitation.

KUN CHAE  
BARBARA J. CORBETT  
PHILLIP W. ALBRO

National Institute of Environmental Health  
Science  
P.O. Box 12233  
Research Triangle Park, N.C. 27709

#### REFERENCES

1. Zitko, V., Hutzinger, O., and Safe, S., Bull. Environ. Contam. Toxicol. 6: 160 (1971).
2. Gregory, N. L. J. Chem. Soc. (B) 1968: 295 (1968).
3. Environ. Health Perspect. No. 1 (1972).
4. Berg, O. W., Diosady, P. L., and Rees, G. A. V. Bull. Environ. Contam. Toxicol. 7: 338 (1972).